

A Comparative Study of the Adsorption of Lead in Aqueous Solution by Raw and Iron-Functionalized Bivalve Shell Powder

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Abstract Water contamination by trace metals (TM), particularly lead (Pb^{2+}), poses a major threat to human health and the environment. In Côte d'Ivoire, the growth of industrial and mining activities is exacerbating this pollution, creating a need for effective and accessible solutions. This study evaluated the effectiveness of raw bivalve shell powders (COQ) and iron-modified bivalve shell powders (COQ-Fe) for the removal of lead from aqueous solution. The lead in solution was analyzed by atomic absorption spectroscopy (AAS). The optimal conditions identified were: 1.5 g of powder per 100 mL, pH 5, and a contact time of 60 minutes. Under these conditions, COQ achieved 98.84% removal, and COQ-Fe achieved over 99% removal. Kinetic analysis showed a fit to the pseudo-second-order model, while the modified Langmuir model confirmed an ion exchange mechanism between Pb^{2+} and Ca^{2+} . These results highlight the potential of bivalve shells as a local, sustainable, and low-cost solution for the remediation of water contaminated with heavy metals.

Keywords: shell powder, lead, decontamination, ion exchange

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1. Introduction

Water pollution caused by trace metals is a major problem due to their toxicity, persistence, and accumulation in living organisms. Primarily resulting from industrial, mining, agricultural, and urban activities, these metals cause long-term contamination of aquatic environments and pose a serious risk to ecosystems and human health. Numerous studies have shown that human activities lead to high levels of metal contamination in water, soil, plants, and sediments in many regions of the world [1,2].

Developing effective and sustainable processes to remove metals from contaminated water is a priority. Several techniques are used, including flocculation-coagulation, chemical precipitation, adsorption, ion exchange, reverse osmosis, and solvent extraction... [3]. Adsorption is the most widely used method for treating contaminated water, with activated carbon and clay serving as traditional adsorbents. Shellfish waste, which is often not recycled, represents an interesting alternative. In

fact, using this waste helps both reduce marine pollution and purify water, thanks to its calcium carbonate content, which is effective against metal cations [4,5,6]. Furthermore, modifying the powder through iron treatment can enhance its ability to remove metal species [7]. This study aims to evaluate the effectiveness of raw and iron-modified oyster shell powder in removing lead from aqueous solutions. The study examines the influence of parameters such as the mass of oyster shell powder, pH, contact time, and initial concentration on the process's performance.

2. Materials and Methods

2.1. Shell

The shells used were collected in the village of Bodou, located 13 km southwest of Dabou, in the Lagunes District of Côte d'Ivoire. This site was chosen for its abundance of shells and ease of access. The geographic coordinates are 5°18'11" N and 4°27'59" W.

2.2. Methods

2.2.1. Sample Preparation

Before use, the shells were washed in cold water to remove impurities, dried at 40°C for 24 hours, and then ground and sieved. Only particles with a diameter of less than 0.2 mm were selected for further analysis. Figure 1 shows the appearance of the raw and ground samples.

The mineral composition and pH at the zero-charge point of the shell powder are presented in Table 1.



Figure 1. Raw and powdered shells

Table 1. Mineralogical composition and pH at the zero-load point (pH_{PCN}) of COQ

Sample	%Calcite	%Quartz	%Others	pH_{PCN}
COQ	81.4	9.81	8.79	8.7

2.2.2. Preparation of an Iron-functionalized Shell Powder (COQ-Fe)

The shell powder was modified by impregnation with iron polycations. To do this, a ferric solution prepared by reacting iron (III) chloride hexahydrate with NaOH under agitation was mixed with a suspension of shell powder in order to incorporate the ferric species into the material.

2.2.3. Preparation of the Iron-based Solution

The iron solution was prepared by slowly titrating 40 mL of an iron(III) chloride hexahydrate solution ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 0.43 M) with 46 mL of a sodium hydroxide solution (NaOH, 0.75 M) under constant magnetic stirring. This titration was performed to maintain a molar ratio of $\text{OH}^-/\text{Fe}^{3+} = 2$, promoting the formation of $\text{Fe}_x(\text{OH})_y$ polycations. The resulting reddish-brown solution had a final iron concentration of 0.2 mol/L. It was then left to stand in the dark for 48 hours to allow the solution to mature.

2.2.4. Treatment of Shell Powder with an Iron Solution

The fine shell powder was modified using a ferric solution ($\text{Fe}_x(\text{OH})_y$). The treatment consisted of adding

this solution drop by drop to a suspension of shell powder that was kept under continuous stirring. The suspension was prepared using 2.45 g of powder in 490 mL of water (0.5% w/v). After the ferric solution had been fully added, stirring was continued for 24 hours to promote the incorporation of the polycations. The resulting powder was then washed with distilled water until the AgNO_3 test was negative, then dried at 70°C and calcined at 450°C for 4 hours. Figure 2 shows the appearance of the iron-treated shell powder (COQ-Fe).



Figure 2. Iron-modified shell powder

2.2.5. Preparation of Lead Solutions

A 1000 mg/L lead stock solution was prepared by dissolving 1.6 g of $\text{Pb}(\text{NO}_3)_2$ (99.99% purity) in 1 L of distilled water. This solution was used to prepare various working solutions at the desired concentrations by dilution.

2.2.6. Lead Removal Reaction

For each experiment, a quantity of shell powder (COQ or COQ-Fe) is placed in contact with lead-contaminated water in a beaker. The shell-solution mixture is stirred for a short time at room temperature. After stirring, the mixture is centrifuged at 3,000 rpm for 15 minutes, and the supernatant is collected for the determination of chemical species using the SAA method to determine the residual lead content in solution. The efficiency of the lead removal process is determined by calculating the sorption

capacity ($Q_t = \frac{C_0 - C_t}{m} V$) and the removal efficiency of

the compound ($R = \frac{C_0 - C_t}{C_0} * 100$). The influence of the

mass of shell powder, the pH of the solution, and the contact time was first examined to determine the optimal conditions for lead removal using raw shell powder (COQ). Subsequently, the efficiency of lead removal was evaluated using both raw shell powder (COQ) and iron-modified shell powder (COQ-Fe), while varying the initial lead concentration. Figure 3 summarizes the lead removal reaction that was carried out.

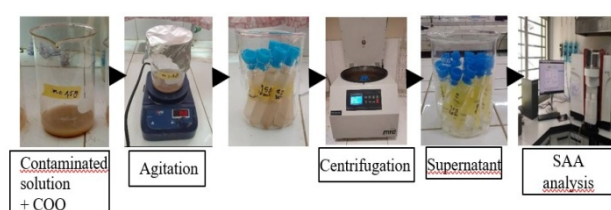


Figure 3. Summary of the study protocol for lead removal

2.2.7. Effect of Mass on Lead Removal

Lead removal was studied as a function of the mass of shell powder. Suspensions containing 100 mL of a 20-ppm lead solution were prepared using different masses of powder (0.5 to 2 g) and stirred for 1 hour at pH 6 to determine the optimal amount of material for decontamination.

2.2.8. Study of the Effect of pH

pH is a key parameter in the study of lead removal. In this study, the effectiveness of raw oyster shell powder (COQ) was evaluated at different pH values. To do this, 100 mL of a 20 ppm lead solution was mixed with 1.5 g of raw oyster shell powder. The pH of the suspensions was adjusted using HCl and NaOH solutions, and lead removal was studied at pH values ranging from 3 to 7.

2.2.9. Study of Contact Time

Lead sorption was monitored over a time range of 5 to 90 minutes, with an initial concentration of $20 \text{ mg} \cdot \text{L}^{-1}$ and 1.5 g of shell powder in 100 mL of solution. The pH of the suspensions was maintained at 5. This study characterizes the kinetics of lead removal and evaluates the material's effectiveness.

2.2.10. Study of the Effect of Initial Pb^{2+} Concentration on the Sorption Capacity of COQ and COQ-Fe

Lead removal was evaluated at various initial concentrations. In a series of beakers, 1.5 g of raw shell powder (COQ) was added and brought into contact with 100 mL of lead solutions whose concentrations ranged from 5 to 30 mg/L, depending on the beaker. The mixture was stirred for 60 minutes at room temperature using magnetic stirrers. The pH of the solutions was maintained at 5. After centrifugation, the supernatants were analyzed. The residual concentrations at equilibrium (C_e) were measured, and the amounts removed per unit mass at equilibrium (Q_e) were determined. This study was also conducted using iron-modified oyster shell powder (COQ-Fe) to evaluate the effect of iron oxides on lead removal efficiency.

3. Results and Discussion

3.1. Effect of Mass on Lead Removal

The results for the percentage of lead removal are shown in Figure 4.

Increasing the mass of shell powder from 0.5 g to 2 g results in a higher percentage of lead removal, with retention rates consistently above 98% and a maximum of 99.76% for 1.5 g. This improvement can be explained by the increased availability of active sites that promote the binding of Pb^{2+} ions, consistent with the results reported by Amimeur [8] and Wu *et al.* [9]. The observation of a plateau starting at 1.5 g suggests that the active ingredients available for lead removal have reached saturation. Based on this study, 1.5 g was selected as the optimal mass for more effective lead removal in subsequent work.

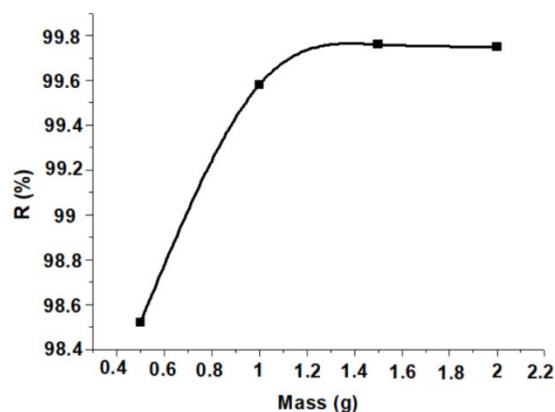


Figure 4. Effect of COQ mass on lead removal

3.2. Effect of Solution pH on Lead Removal

The pH of the solution is a key factor in lead removal processes. The results showing the effect of pH on decontamination are presented in Figure 5.

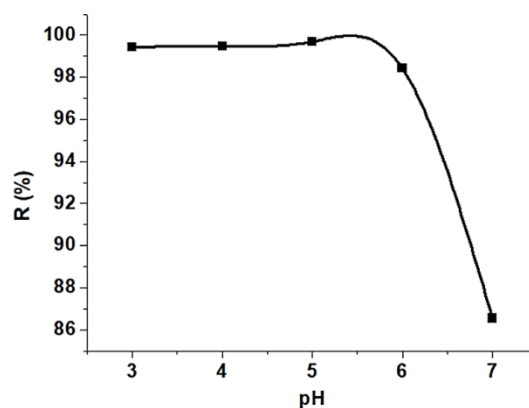


Figure 5. Effect of pH on Lead Removal

For different pH values, the percentage of lead removal varies to a greater or lesser extent. When the pH is between 3 and 5, a pseudo-plateau is observed, with removal efficiencies exceeding 99%. The lead removal rate peaks at pH = 5 (99.69%). At pH values above 5, the removal rate gradually decreases until it reaches 86.56%.

This observation can be attributed to the high capacity of shell powder to effectively bind Pb^{2+} ions in an acidic environment. This behavior is confirmed by Akou's research [10], which reported zinc removal rates ranging from 94% to 99% for initial pH values of 3 to 6, as well as by Lee *et al.* [11] who achieved removal rates close to 99% for lead using crab shell particles within a pH range of 3 to 7.

Between pH 6 and 7, a decrease in efficiency is observed due to the precipitation of lead as hydroxide ($\text{Pb}(\text{OH})_2$). Indeed, the Pb^{2+} ion remains the dominant species below pH 6, whereas above that value, the precipitated form $\text{Pb}(\text{OH})_2$ becomes predominant (Figure 6). This phenomenon was also demonstrated by Sedira [4], which showed that the adsorption of heavy metals on activated carbon derived from date pits is strongly influenced by pH, with a significant decrease when the precipitation of metal ions becomes thermodynamically favorable. Similarly, Mimanne *et al.* [12] reported that the

adsorption of lead and cadmium peaks within an optimal pH range, after which it decreases due to the formation of metal precipitates. At pH values above 6, $\text{Pb}(\text{OH})_2$ species which are larger than Pb^{2+} ions limit the efficiency of ion exchange with the Ca^{2+} ions present in the shell powder, thereby reducing the percentage of lead removed. In light of the above, lead retention is optimal at pH 5.

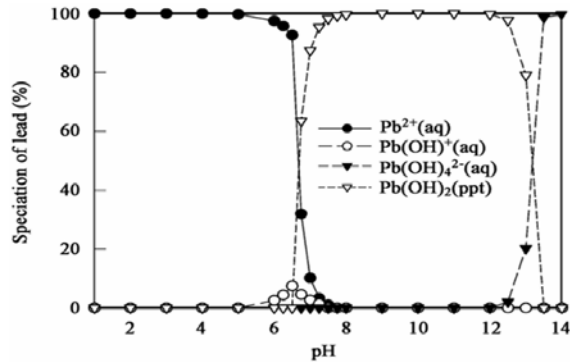


Figure 6. Speciation of lead ions (3 mg/L) in an aqueous solution

3.3. Effect of Contact Time

The results of lead removal as a function of agitation time are shown in Figure 7.

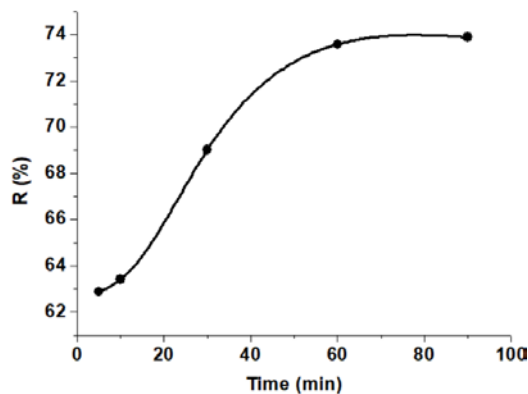


Figure 7. Lead removal kinetics

According to the results obtained, lead removal progresses rapidly from the very first minutes of contact, reaching a retention rate of 62.88% after only 5 minutes of agitation. This trend continues as contact time increases, until a pseudo-plateau is reached starting at 60 minutes, at which point the recovery rate exceeds 73%. The rapid removal of lead observed at the start of the process is characteristic of the physicochemical interactions between metal ions and the shell powder. These interactions, which require little energy, are based on phenomena such as electrostatic attraction, hydrogen bonding, and Van der Waals forces, allowing for effective binding of the ions from the very first moments of contact [13]. The abundant availability of functional sites on the surface of the biosorbent also promotes this initial adsorption. As the contact time increases, these sites gradually become saturated, leading to a decrease in the adsorption rate [8].

The equilibrium time can be estimated at 60 minutes for the entire process.

3.4. Modeling the Kinetics of Lead Removal

The results of the elimination kinetics were modeled using the assumptions of pseudo-first-order (Figure 8) and pseudo-second-order (Figure 9) models. The equations used for these models are as follows: $\frac{dQ_t}{dt} = K_1(Q_e - Q_t)$

and $\frac{dQ_t}{dt} = K_2(Q_e - Q_t)^2$ with K_1 : first-order rate constant (min^{-1}); Q_t : adsorption capacity at time t in mg/g ; Q_e : equilibrium adsorption capacity in mg/g ; and K_2 : pseudo-second-order adsorption rate constant ($\text{g} \cdot \text{mol}^{-1} \cdot \text{min}^{-1}$ or $\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$). The correlation coefficient values and the experimental and theoretical adsorption capacities determined by the two models are summarized in Table 2.

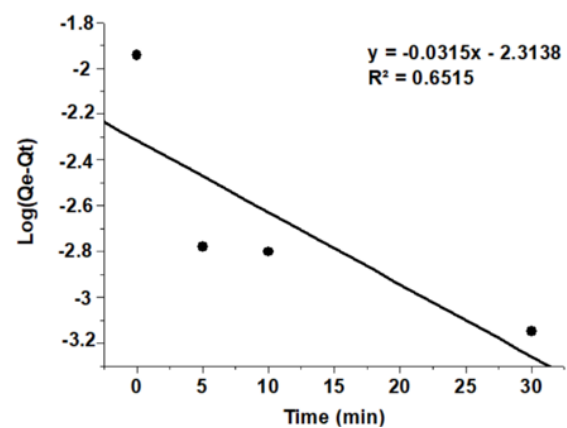


Figure 8. Application of the pseudo-first-order kinetic model to lead removal

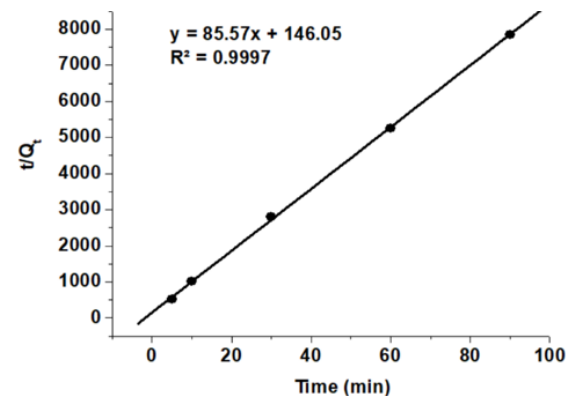


Figure 9. Application of the pseudo-second-order kinetic model to lead removal

Table 2. Values of correlation coefficients and parameters obtained from the pseudo-first-order and pseudo-second-order kinetic models

Pseudo-first-order kinetics			Pseudo-second-order kinetics			Q_e (mg/g) experimental
Q_e (mg/g) theoretical	K_1 (min^{-1})	R^2	Q_e (mg/g) theoretical	K_2 ($\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$)	R^2	
0.0049	0.0315	0.6515	0.0117	50.018	0.9997	0.0114

The theoretical removal capacity calculated using the pseudo-second-order kinetic model (0.0117 mg/g) is very

close to the experimental value (0.0114 mg/g). This model also exhibits an excellent coefficient of determination ($R^2 \geq 0.99$), unlike the pseudo-first-order model, which has a low R^2 (0.6515). Furthermore, the rate constant obtained with the pseudo-second-order model ($K_2 = 50.018$) is more than 1,500 times greater than that of the pseudo-first-order model ($K_1 = 0.0315$). These results show that the retention of Pb^{2+} ions by shell powder follows the pseudo-second-order model perfectly, suggesting a mechanism dominated by chemisorption. This conclusion is consistent with the work of Bozbaş and Boz, who observed similar behavior for the biosorption of $Pb(II)$ and $Cu(II)$ ions on the shells of the gastropod *Rapana venosa* [14].

3.5. Effect of Initial Lead Concentration on the COQ and COQ-Fe Retention Capacity

Figure 10 shows the variation in the sorption capacity of raw oyster shell powder (COQ) and iron-treated oyster shell powder (COQ-Fe) as a function of the initial lead concentration. The calcium concentrations before ($[Ca^{2+}]_i$) and after ($[Ca^{2+}]_f$) the sorption reaction with COQ are also presented in Table 3.

An increase in the initial lead concentration (0–30 ppm) results in a significant increase in the amount removed. Raw shell powder (COQ) exhibits a retention capacity ranging from 0.329 to 1.940 mg/g, with a removal rate of up to 98.84%. The concentration of Ca^{2+} ions in solution increases from 1.845 mg/L before the reaction to more than 79 mg/L after sorption. The iron-modified powder (COQ-Fe) exhibits slightly superior performance, with a capacity ranging from 0.322 to 1.986 mg/g and lead removal exceeding 99%. The sorption isotherms plotted (Figure 11 and Figure 12) show a similar trend for both materials, characterized by two pseudo-plateaus.

Table 3. Lead sorption as a function of initial concentration on COQ and COQ-Fe

C_0 (mg/L)	COQ					COQ-Fe				
	5	10	15	20	30	5	10	15	20	30
C_e (mg/L)	0.058	0.400	0.473	0.810	0.900	0.175	0.171	0.184	0.146	0.213
Q_e (mg/g)	0.329	0.640	0.968	1.279	1.940	0.322	0.655	0.988	1.324	1.986
R (%)	98.84	96	96.85	95.95	97	96.51	98.29	98.77	99.27	99.29
$[Ca^{2+}]_i$ (mg/L)	1.845									
$[Ca^{2+}]_f$ (mg/L)	79.25	79.81	82.93	88.41	87.17					

The efficiency of lead removal by COQ and COQ-Fe increases with the initial concentration, because a stronger gradient promotes the diffusion and binding of Pb^{2+} ions to the material's active sites [15]. The high sorption rates obtained demonstrate that the shell powder used in this study is a particularly effective material for the remediation of lead-contaminated solutions. Similarly, numerous studies have confirmed the remarkable effectiveness of calcium carbonate derived from shells in the treatment of wastewater containing metal cations [11,16,17]. The slight improvement in the lead removal rate observed with the COQ-Fe sample can be attributed to the presence of iron in the shell powder. Indeed, several studies have shown that incorporating iron into materials enhances their effectiveness, creating matrices that are

particularly effective at removing trace metals [18,19]. According to the classification proposed by Giles and his colleagues, the isotherms obtained in this study belong to type S. This profile reflects a variable affinity between the shell powder and lead ions, which is particularly pronounced at low concentrations [18].

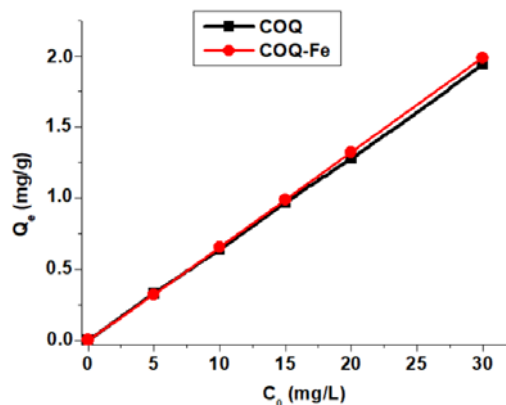


Figure 10. Effect of Initial Concentration on Lead Removal

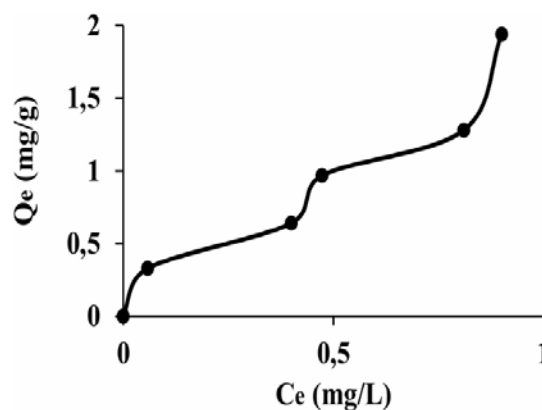


Figure 11. Sorption isotherm for lead on COQ

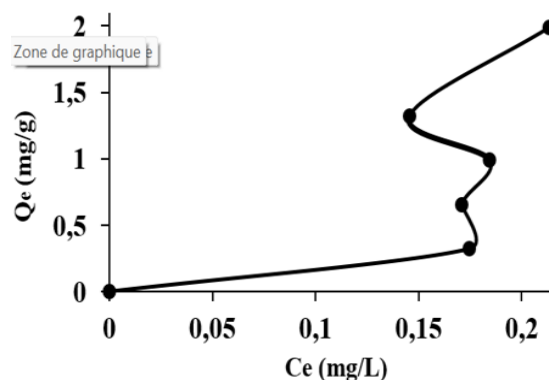


Figure 12. Isotherm for lead sorption on COQ-Fe

3.6. Application of Linear Adsorption Models to Isotherms

The results regarding lead removal were modeled based on the assumptions of Langmuir's mathematical models ($\frac{C_e}{Q_e} = \frac{1}{Q_{\max}} C_e + \frac{1}{Q_{\max} K_L}$), Freundlich ($\log Q_e = \log K_f + \frac{1}{n} \log C_e$), Temkin ($Q_e = B \ln K_T + B \ln C_e$), Dubinin-

Radushkevitch ($\ln Q_e = \ln Q_m^{\text{DR}} - \beta \varepsilon^2$) and Elovich ($\ln \frac{Q_e}{C_e} = \ln K_e Q_m - \frac{Q_e}{Q_m}$) [20].

The correlation coefficients obtained from the various models were calculated and summarized in Table 3.

Table 3. Correlation coefficients based on the Freundlich, Langmuir, Temkin, Dubinin-Radushkevich, and Elovich models

R ²	COQ	COQ-Fe
Freundlich Model	0.8795	0.0691
Langmuir Model	0.4127	0.0126
Temkin Model	0.6762	0.1627
Dubinin-Radushkevich Model	0.7536	0.0523
Modèle de Elovich	0.2386	0.831

The linear adsorption models tested exhibit insufficient correlation coefficients ($R^2 < 0.90$), indicating that they do not accurately describe the lead removal process. This observation suggests that the mechanism at work in the oyster shell powder is not solely a matter of adsorption. It therefore appears necessary to use more appropriate models to accurately represent the observed removal mechanism.

3.7. Application of the Modified Langmuir Model to Ion Exchange in Sorption Isotherms

When dealing with a binary ion-exchange system, it is more appropriate to use a derived formulation known as the modified Langmuir model. This approach accounts for the competition between the two cations for exchange sites and expresses the relationship between the equivalent fractions in the reactive solid and in the solution [21,22]. This approach is illustrated by the following relationship :

$$\frac{\chi_i}{1-\chi_i} = K_{i/j} \frac{C_i}{C_j} \quad \text{where: } \chi_i : \text{Fraction of metal ions}$$

occupying the solid phase; $K_{i/j}$: Ion exchange constant ;

$K_{i/j}$ reflects the sorbent's capacity to retain the metal ion and is a key indicator of the efficiency of the ion exchange

process [21]. Plotting the line $\frac{\chi_i}{1-\chi_i} = f\left(\frac{C_i}{C_j}\right)$ allows the

slope $K_{i/j}$ to be determined.

Figure 13 illustrates the fit of the modified Langmuir model applied to the ion exchange mechanism to the lead sorption isotherm on raw oyster shell powder.

Lead removal by the COQ sample is better described by the modified Langmuir model applied to the ion exchange mechanism. In fact, the correlation coefficient obtained is greater than 0.9, while the ion exchange constant ($K_{\text{Pb/Ca}}$) is significantly greater than 1 (183.11). This result confirms that lead removal is not simply a matter of uniform monolayer adsorption. Other mechanisms may be involved, such as precipitation, ion exchange, or specific chemical reactions with the constituents of the shell powder, particularly calcium carbonate. According to Papadopoulos et al., at low concentrations, Cd^{2+} cations

can substitute for Ca^{2+} ions present at accessible surface sites ($\text{Cd}^{2+} + \text{CaCO}_3 \rightarrow \text{CdCO}_3 + \text{Ca}^{2+}$). Once these sites are saturated with Cd^{2+} ions, the formation of CdCO_3 continues through a nucleation process, followed by precipitation on the surface of CaCO_3 , according to the following reaction : $\text{Cd}^{2+} + \text{HCO}_3^- \rightarrow \text{CdCO}_3 + \text{H}^+$ [23]. The high value of the ion exchange constant ($K_{\text{Pb/Ca}} = 183.11$), significantly greater than one, confirms that lead removal occurs primarily through an ion-exchange mechanism, involving the replacement of Ca^{2+} ions present in the shell matrix with Pb^{2+} ions.

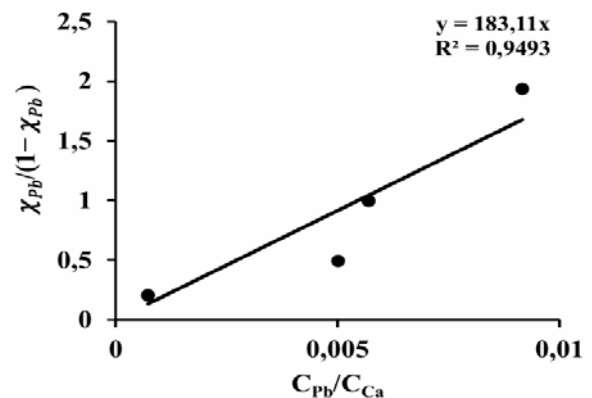


Figure 13. Application of the modified Langmuir model to the ion exchange mechanism

4. Conclusion

The purpose of this study was to evaluate the effectiveness of raw shell powder (COQ) and iron-modified shell powder (COQ-Fe) in removing lead (Pb^{2+}) from aqueous solution. A powder enriched with iron polycations was synthesized from the raw powder to enhance its ability to remove lead. The influence of several physicochemical parameters, such as the mass used, the solution pH, and the contact time, was examined. The optimal conditions identified were: 1.5 g of powder per 100 mL of solution, a pH of 5, and a contact time of 60 minutes, resulting in maximum lead removal.

Under these conditions, the effect of the initial lead concentration (5 to 30 mg/L) was studied for both materials. The raw powder achieved a removal rate of up to 98.84%, while the iron-modified version exceeded 99%. Kinetic analysis revealed that the process follows a pseudo-second-order model, reflecting a gradual interaction between Pb^{2+} ions and the material's active sites.

Conventional adsorption models (Langmuir, Freundlich, Temkin, Dubinin-Radushkevich, Elovich) failed to accurately describe the observed mechanism. In contrast, the modified Langmuir model, when applied to the ion exchange mechanism, proved to be more relevant. The high ion exchange constant ($K_{\text{(Pb/Ca)}} = 183.11$) confirms that lead is primarily removed by substitution of Ca^{2+} ions in the carbonate matrix.

These results highlight the potential of oyster shell powder as an effective material for the remediation of lead-contaminated water.

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